

16:16:02

OCA PAD INITIATION - PROJECT HEADER INFORMATION

08/11/88

Project #: [REDACTED]  
Center #: R6524-OAO

Cost share #: G-33-333  
Center shr #: F6524-OAO

Rev #: 0  
OCA file #:  
Work type : RES  
Document : GRANT  
Contract entity: GTRC

Active

Contract #: [REDACTED]  
Prime #: [REDACTED]

Mod #:

Subprojects ? : N  
Main project #:

Project unit: CHEM Unit code: 02.010.136  
Project director(s): [REDACTED]

Sponsor/division names: [REDACTED]  
Sponsor/division codes: [REDACTED]

Award period: [REDACTED] (reports)

Sponsor amount	New this change	Total to date
Contract value	164,888.00	164,888.00
Funded	164,888.00	164,888.00
Cost sharing amount		14,700.00

Does subcontracting plan apply ? : N

Title: [REDACTED]

PROJECT ADMINISTRATION DATA

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Sponsor technical contact

Sponsor issuing office

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Security class (U,C,S,TS) :

ONR resident rep. is ACO (Y/N): N  
supplemental sheet

Defense priority rating :

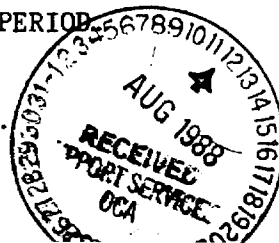
Equipment title vests with: Sponsor

GIT X

NO EQUIPMENT MAY BE PURCHASED IN LAST SIX MONTHS OF APPROVED GRANT PERIOD

Administrative comments -

INITIATION. 1ST YEAR OF A GRANT RECOMMENDED FOR SUPPORT FOR 3 YEARS.



X   Reports Coordinator (OCA)  
  X   GTRC  
  X   Project File  
    2   Contract Support Division (OCA)  
       Other

SECTION IV PROGRESS REPORT SUMMARY		GRANT NUMBER 1 RO1 GM40715-02	
PRINCIPAL INVESTIGATOR OR PROGRAM DIRECTOR Richard F. Browner		PERIOD COVERED BY THIS REPORT	
APPLICANT ORGANIZATION Georgia Institute of Technology		FROM 7.1.88	THROUGH 6.30.89
TITLE OF PROJECT (Repeat title shown in item 1 on first page) FUNDAMENTAL STUDIES FOR BIOANALYTICAL CHEMISTRY (SEE INSTRUCTIONS)			

## 1. Planned Research.

This project is for the development of a liquid chromatography/Fourier transform infrared spectrometric interface. The interface, called a MAGIC-LC/FT-IR spectrometric interface, has had considerable success removing liquid chromatographic mobile phases from solutes so that the solutes can be investigated using infrared spectrometry. The second year of the research project will focus on measurements on the particle beam itself. The first year of the project provided funds for the purchase of a laser photon correlation anemometer to study the particle densities and velocities of the particle beam in the monodisperse aerosol generator. This equipment has been delivered and the studies are currently being set up. The specific parameters to be measured are particle density, particle velocity and particle trajectory maps throughout the interface. These data will provide detailed information on the characteristics of the interface that can be used to improve the overall efficiency of the analysis technique. The anemometry studies will also provide information on the effects of varying experimental conditions, such as liquid chromatographic flow rate and dispersion gas pressure and flow rate. All these parameters will provide data for the improvement of the interface transport efficiency. These studies will be carried out at the Georgia Institute of Technology.

Research at the University of Georgia will focus on the development of a working interface for MAGIC-LC/FT-IR spectrometry. All studies thus far have been accomplished "off-line". That is, effluent from a chromatograph has been passed into a MAGIC-LC device and the solutes have been collected onto infrared transparent windows. The windows have been moved to an FT-IR spectrometer where spectra were recorded. The two steps are to be combined to make a MAGIC-LC/FT-IR interface. This will involve optics to focus the infrared radiation onto the deposited solutes. Mechanical translation equipment will be used to move the infrared transmitting windows into the infrared beam as the deposition of the solute and the interrogation of the solute cannot be accomplished at the same location. In addition to the investigation of deposited solutes, studies will be initiated for the investigation of "on-the-fly" analyses. That is, a cell will be constructed whereby the particle beam will be interrogated with an infrared beam. If spectra can be recorded using this on-the-fly method the MAGIC-LC/FT-IR device can be used in conjunction with other interfaces, such as MAGIC-LC mass spectrometry. All interface optical systems will be carried out with relatively large samples as optics for microscopic sampling were not provided in this award.

All the studies outlined are as presented in the original proposal, and there is no change to the overall plan, except that the microscopic studies were not funded and these cannot be pursued as equipment does not exist at either site.

## 2. Research Performed During Current Budget Year.

Research has focussed on the characteristics of the MAGIC-LC/FT-IR spectrometric device. Two primary studies have been completed, one on the use of buffers in the liquid chromatographic mobile phase and the effect on detection of solutes, and the other on transportation efficiencies of the device.

In the first of these studies, the initial purpose was to test the efficiency of removal of volatile buffers as well as solvent from the chromatographic effluent stream. If volatile buffers could be removed from the effluent stream and leave no infrared detectable trace, then buffered separations could be accomplished. There has been success using these buffers, for example ammonium acetate, in MAGIC-LC mass spectrometric studies. Removal of volatile buffers in the infrared studies has not been as successful as the buffer tends to be trapped in the solute depositions, yet no volatile buffer is deposited on the deposition window when no solute is present in the effluent stream. Clearly this is a matrix effect. As the buffers were deposited with a solute, studies with involatile buffers were initiated, specifically with potassium dihydrogen phosphate and potassium hydrogen phthalate buffers. In these instances the buffer was deposited regardless of whether a solute was present. Spectral subtraction was used to obtain the spectrum of the solute by subtracting a spectrum of the buffer plus solute from a spectrum of the buffer alone. These studies were carried out in a variety of mobile phases, including aqueous mobile phases such as acetonitrile:water. The implications of this study are that MAGIC-LC/FT-IR spectrometry is successful and independent of chromatographic mobile phase composition. A manuscript describing this work will be submitted in the near future.

The second study has involved transport efficiencies in the MAGIC-LC/FT-IR spectrometric interface. This device is different from the MAGIC-LC mass spectrometric device reported in the initial proposal, in as much the pressure requirements are not as stringent. It has been found that the efficiency of the MAGIC-LC/FT-IR spectrometric device is on the order of 25%. That is, approximately 25% of any solute injected into a liquid chromatograph is deposited onto the window for infrared investigation. This study is on-going and will be continued as data are provided from the laser correlation anemometry studies outlined in the proposed work for year two.

All other tasks outlined in the original proposal, such as software design, have been completed.

### 3. Research Involving Human Subjects.

Not applicable.

### 4. Research Involving Vertebrate Animals.

Not applicable.

### 5. Publications.

James A. de Haseth and Raymond M. Robertson, "MAGIC-LC/FT-IR: A Viable Interface for HPLC and FT-IR Spectrometry", *Microchemical Journal*, 1989, in press.